PATENT SPECIFICATION

NO DRAWINGS

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Int. CL:-C 08 f 29/18, C 08 f 45/58

COMPLETE SPECIFICATION

Heat Stabilized Vinyl Chloride Polymer Compositions

We, W. R. GRACE & Co., a Corporation organized and existing under the laws of the State of Connecticut, United States of America, of 7, Hanover Square, New York 5, New York, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the stabilization of vinyl chloride homopolymers

and copolymers against heat.

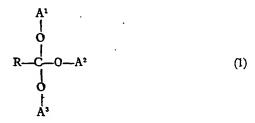
It is well known that vinyl chloride containing resins degrade at elevated temperatures. When vinyl chloride homopolymers and copolymers are moulded at temperatures in excess of about 150°C, they tend to discolour. Serious discoloration occurs even in the relatively short time required for a moulding operation.

Numerous stabilizers have hitherto been suggested for use in vinyl chloride type resins. The most satisfactory of these stabilizers are tin, lead and cadmium compounds. These compounds, while performing satisfactorily where toxicity is not a problem, cannot be used if the treated polymer is likely to come into contact with foodstuffs.

In our Specification No. 1,110,958 we have described and claimed heat stabilized compositions which comprise vinyl chloride homopolymers or copolymers and, as stabilizer, an ortho ester of the formula R_1 — $C(OR)_s$, wherein R_1 represents an alkyl radical of 6 to 20 carbon atoms, or a phenyl group substituted by halogen or nitro and R represents an alkyl group of 1 to 12 carbon atoms; or of the formula $X-[-R_2-C(OR)_3]_2$ wherein X represents an oxygen or sulphur atom, R_2 is an alkylene group of 1 to 4 carbon atoms, and R represents an alkyl group of 1 to 12 carbon atoms. These stabilizers are non-toxic and may be incorporated in resins which are to be used in the packaging of foodstuff materials. We have now found that certain other compounds may be used as stabilizers for vinyl chloride homopolymers and copolymers which are also non-toxic and may be used in resins which may be incorporated in resins which are to be used in the packaging of foodstuff materials.

The present invention, which is an improvement in or modification of the invention of Specification No. 1,110,958, accordingly provides heat stabilized compositions which comprise vinly chloride homopolymers or copolymers and, as stabilizer, at least one

compound of formula:



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wherein R represents an alkyl, phenyl, phenylalkyl, alkylphenylalkyl, halophenyl, nitrophenyl or alkenyl radical and A', A' and A', which may be the same or different, are hydrogen radicals or oxygen-interrupted hydrocarbon radicals and wherein any two or all of A', A' and A' may be joined together to form a ring or rings or of formula

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wherein R¹, R² and R², which may be the same or different, represent alkyl, phenyl, phenylalkyl alkylphenyl, epoxyalkyl or alkylphenylalkyl radicals, R⁴ represents an alkylene, phenylene or alkylphenylene radical and X represents an oxygen or sulphur atom or a single bond. The amount of stabilizer in the compositions is preferably from 1 to 10% by weight based on the weight of the polymer. Desirably the ortho esters used should form a homogeneous blend with the polymer at the processing temperature. Preferred ortho esters are those having a low vapour pressure at the processing temperature since excessive foaming of the resin is thereby avoided.

Particularly suitable orther esters are:

(3) those of formula (1) above wherein A¹, A² and A³ represent alkyl, phenyl, phenylalkyl, alkylphenyl, epoxyalkyl or alkylphenylalkyl radicals.

(4) those of formula (2) above, and those of the following formulae:

wherein R is as defined in formula (1) above, A¹ and A² are as defined in (3) above; R³ represents an alkylene, phenylene, alkylalkylene, alkenylene, alkenyl oxyalkylalkylene or alkynylene radical; and n is an integer of from 1 to 4.

$$\begin{array}{c} 0 - R^{6} \\ R - C - 0 \\ 0 A^{1} \end{array}$$

wherein R is as defined in formula (1) above, A¹ is as defined in (3) above; and R⁶ represents an alkylene, alkylalkylene or alkenylalkoxyalkylene radical.

(7) wherein R is as defined in formula (1) above, and R⁵, R⁶ and n are as defined in (5) and (6) above.

$$R-C-O-R^{7}-C-R^{8}$$

wherein R is as defined in formula (1) above, R' represents an alkylene radical, and R' has any of the meanings given for R in formula (1) above.

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wherein y is an integer of from 2 to 10.

The following three methods may be used to prepare orthoesters:

A. Iminoester route, as set forth in Pinner, Ber., 16 356, 1644 (1883).

The appropriate nitrile is reacted with one equivalent of dry hydrogen chloride and one equivalent of alcohol to form an iminoester hydrochloride which is then alcoholyzed with an excess of alcohol to form the orthoester. The reaction may be represented as follows:

NH.HCl excess R'OH RCN+R'OH+HCl \rightarrow RCOR'+ \longrightarrow RC(OR')_a+NH₄Cl

10 A dinitrile can be used as follows:

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NH₂Cl NH₂Cl excess

|| || R'OH

NCR'CN+2R'OH+2HCl \rightarrow R'OC—R—COR'—— \rightarrow (R'O)₃CRC(OR')₃+2NH₄Cl

Furthermore, diols may be used at either one or both steps of the synthesis.

$$\begin{array}{c} \text{NH. HCl} & \text{NH. HCl} \\ \text{2RCN} + \text{HOR}^{\text{I}}\text{OH} + \text{2HCl} \xrightarrow{\text{RCO}-\text{R}^{\text{I}}} \text{RCO}^{\text{I}} - \text{RCO}^{\text{I}} \\ \text{RCO}^{\text{I}} & \text{RCO}^{\text{I}} - \text{RCO}^{\text{I}} \\ \text{RCO}^{\text{I}} & \text{RCO}^{\text{I}} & \text{RCO}^{\text{I}} \\ \text{RCO}^{\text{I}} & \text{RCO}^{\text{I}} \\ \text{RCO}^{\text{I}} & \text{RCO}^{\text{I}} & \text{RCO}^{\text{I}} \\ \text{RCO}^{\text{I}} &$$

B. Exchange reaction as described by Mkhitaryan, V.J. Gen. Chem. (USSR) 8 1361 (1938).

The alkoxy groups of a readily available orthoester such as triethyl orthoacetate are displaced by a higher boiling alcohol or polyol as follows:

 $CH_3C(OC_2H_5)_3 + 3ROH \rightarrow CH_3C(OR)_3 + 3C_2H_5OH$

Furthermore, the reaction may be carried out in two steps with two different alcohols or polyols.

C. Alcoholysis of trihalomethyl groups as described by Sah, P. and Ma., S.T., J. Am. Chem. Soc. 54, 2664 (1932).

The appropriately substituted trichloromethyl compound is treated with a metal alkoxide.

 $RCCl_3+3NaOR'\rightarrow RC(OR')_3+3NaCl$

Specific examples of orthoesters which may be used in the compositions of the

present invention are given in the following paragraphs along with a general description f how they may be prepared. The numeral designations will be used in the subsequent specific Examples to identify these compounds.)

I

This compound is conveniently prepared by routes A or B.

II
$$CH_3 - CC_2H_5 - CC_2H_5$$
 $CC_2H_5 - CC_2H_5$
 $CC_2H_5 - CC_2H_5$

This compound is prepared by routes A or B.

$$OC_2H_3$$
 H H OC_2H_3 OC_2H_3 OC_2H_3 OC_2H_3

Ш

This compound is prepared by routes A or B.

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This compound is prepared by routes A or B.

This compound is prepared by routes A or B.

15 This compound is prepared by routes A or B.

VII
$$CH_2 - C = 0 - CH_2 - C - CH_2 - CH_3$$

This compound is prepared by route A.

This compound is prepared by route B.

(CH₃CH₂CH₂O-)-₃C-(-CH₂-)-₄C-(-OCH₂CH₂CH₃)₃

5 This compound is prepared by routes A or B.

This compound is prepared by route B.

This compound is prepared by routes A and B. XII. Compounds containing the grouping

wherein n is an integer of from 2 to 10. This compound is prepared by route B

 $CH_3C(OC_2H_5)_2$

XIII
This compound is prepared by route A.

CH₃C(OCH₂(CH₂)₆CH₃)₂ | OCH₃

XIV

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This compound is prepared by route A.

(CH₃O)₃C(CH₂)₄C(OCH₃)₅

XV This compound is prepared by route A.

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$(C_2H_3O)_2C(CH_2)_4C(OC_2H_5)_3$

XVI

This compound is prepared by route A.

XVII

This compound is prepared by route B.

XVII

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This compound is prepared by route B.

This compound may be prepared by route B.

It has also been found that the effectiveness of the orthoester stabilizers may be enhanced by adding thereto from 0.1 to 10 moles of a high boiling alcohol per mole

Typical alcohols which may be used possess the general structure

R'(OH),

wherein R' is an organic radical and x is an integer of from 1 to 6. Preferably these alcohols have a boiling point above about 175°C.

In the above formula R' may be alkyl, alkylphenyl, phenylalkyl, alkylene, phenylene, polyalkoxyalkylene and multivalent counterparts thereof. Alcohols which may be used in clude o, m, p-xylene α , α' -diol, trimethylolpropane monopropyl ether, trimethylolpropane monoallyl ether, propylene glycol, diethylene glycol, dimethyloctadiynediol, pentaerythritol, trimethylolpropane, neopentylglycol, benzyl alcohol and dipentaerythritol.

Polyvinyl chlorides which may be heat stabilized in accordance with the invention are vinyl chloride homopolymers and copolymers having a number average molecular weight from about 10,000 to about 150,000 and a weight average molecular weight of from about 20,000 to 1,000,000. These vinyl chloride homopolymers and copolymers are well known. The vinyl chloride copolymers are prepared by copolymerizing vinyl chloride with a copolymerizable monomer such as unsaturated esters which include vinyl acetate, vinyl formate, vinyl benzoate, vinyl stearate and vinyl oleate, as well as diethyl maleate and diethyl fumarate. Copolymers may also be prepared by copolymerizing vinyl chloride with an acrylic ester such as methyl-, ethyl-, butyl- and octyl acrylate. Vinyl chloride copolymers may also be prepared by polymerizing vinyl chloride with vinylidene chloride. The above-mentioned copolymers may contain up to 20% or even 40% by weight of copolymerizing monomer.

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5	The stabilizers are incorporated in the vinyl chloride homopolymers and copolymers by any conventional means. The incorporation may be carried out conveniently by first preparing a slurry of finely divided vinyl chloride homopolymer or copolymer in a solution of the orthoester in a solvent such as methanol, acetone or ethyl ether. The solvent is then separated from the slurry and the polymer particles are dried. This results in polymer particles which are thoroughly coated with the ortho esters. The incorporation of the stabilizers may also be brought about by milling the polymer at the softening temperatures therefor until an intimate blend of the stabilizer with the polymer is achieved. Milling is generally conducted at conventional temperatures of 150 to 200°C. for a time sufficient to obtain a thorough blending of the	5
10	stabilizer with the polymer. The stabilized vinyl chloride compositions may be used in the formation of rigid polyvinyl chloride moulded articles. These rigid moulded pieces are formed in conventional extrusion and injection moulding devices which generally operate at tempera-	10
15	tures of 150 to 200°C. The orthoester stabilizers effectively stabilize the vinyl chloride homopolymer and copolymer during the moulding process and make it possible to produce rigid mouldings having a low degree of colour change and of good clarity. The polyvinyl chloride resins stabilized in accordance with the invention may be mixed with various plasticizers, such as high boiling esters including the alkyl	15
20	phthalates, phosphates, adipates, sebacates and azelates, and with various polymeric ester plasticizers. Also the compositions may contain other additives, such as calcium, zinc, magnesium and tin salts of carboxylic acids, and phosphate esters. Furthermore, the resins may be included in plastisol type preparations which are fabricated by dipping and deposit type moulding techniques.	20
25	The following Examples serve to illustrate the invention.	25
	Examples In the following runs particulate polyvinyl chloride having a number average molecular weight of about 38,000 was dry blended with various amounts of ortho ester and/or polyhydric alcohol. The samples were then placed in the mixing chamber of a	•
30	Brabender Plastograph at 190°C. and open to the air. A roller speed of 60 rpm was used to knead the polymer formulations. The Brabender Plastograph continuously records the torque required to knead the mass. From the torque values it is possible to determine:	30
35	a. The time required for the powder mixture to form a workable plastic mass (flux time) b. The force required to work the plastic mass (average torque value) c. The onset of crosslinking (decomposition time). The actual temperature of the plastic mass was also continuously measured. Small samples of the polymers were	35
40	also removed periodically from the mixing chamber and their colour values compared to those of the standard Gardner scale. The results are summarized in the Table.	40

				TABLE							
	Stabiliser (numerals refer to the ortho	Concentra- tion (parts		Decomposition Time	1	Polymer	Colo	ur (Gardr	ner Scale	Colour (Gardner Scale 0 = Colourless 15 = Brown)	ourless wn)
Run	esters described previously)	per hundred of polymer)	Time Min.	after Flux (min.)	Torque (Kg)	Temp. (°C.)	2 min.	4 min.	10 min.	15 min.	20 min.
1	Λ	5.0	2.0	20.0	1.25	199—204	1.5	1.8	1.5	1.5	1.5 to
7	V m-xylene α,α'-diol	5.0	1.5	26.0	1.25	193—199	0.2	0.2	0.5	1.0	1.2, 1.5 after 25 m.
6	Λ	5.0	2.0	17.0	1.3	195—208	1.0	1.0	1.0	1.5	
	p -xylene α, α' -diol	2.0									
4	V σ-xylene-α,α' diol	5.0	1.5	22.0	1.1	190198	1.5	1.5	1.5	1.5	1.5, blk at 23.5 m.
rv	m-xylene $lpha, lpha'$ -diol	2.0	3.5	10 to 13.5	1.5	190-210	1.5	>15 ·	black		
9	p-yxlene a,a'-diol	2.0	3.5	3.0 to 8.0	1.9	190210	1.5	>15	black		
7	o-xylene α,α'-diol	2.0	2.5	3.5	1.9	190-198 >15	>15	black			
œ	>	5.0	1.5	34.0	4.5	190—197	1.0	2.0	2.0	2.0	2.0, 2.0
	trimethylol propane monoallyl ether (TMPA)	2.0			7:1						200
0	TMPA	2.0	1.0	3.0	1.9	180—194	black				
10	>	5.0	2.0	14.5	1.3	190—203	1.5	1.5	1.5	1.5	
11	V 1,2-propylene glycol	2.0	2.0	12.5	1.4	190200	1.0	1.0	1.0	•	,,
12	>	5.0	2.5	17.0	1.8	190—202	1.0	1.0	1.0	1.0	
	diethylene glycol	2:0	ŧ		?					;	i

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,	lourless own)	20 min.	2.0, 2.0 at 25 m.	black	1.5, 1.5	מונכז כז זו	2.5, 3.0 after 25 m.	3.5 after							. 10	0.5
	0 = 0.	15 min.	2.0	1.8	1.5		2.0								15 15	0.5
	Colour (Gardner Scale 0 = Colourless 15 = Brown)	10 min.	2.0	2.0	1.5		1.5			>15	>15 after flux	>15	1.0		10.5	0.5
	ur (Gard	4 min.	1.5	2.0	1.5		1.0			1.0	2.0 > after flux	1.5	1.0	2.0	3.0	6.0
	Color	2 min.	1.5	2.0	1.5		1.0			1.0	1.0 after flux	1.5	1.0	2.2	2.2	0.5
	Polymer Temp	(°C.)	194—207	194-207	190—198		190—196			190—198	160—192	190—214	189—196	180—197	181—210	178—200
	Torotte	(Kg)	1.3	1.4	1.1		1.1			1.4	1.4	1.4	1.4	1.8	1.8	1.3
	Decompos-		25.0	19	27		31			7.5	8.5	12.0	8.5	8.5	1221 (ill defined)	22.0
	Flux	Min.	3.0	2.5	2.0		2.0			2.0	0.6	2.0	2.0	1.0	1.0	1.0
	Concentra- tion (parts	of polymer)	2.0	5.0	5.0	2.0	5.0		2.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
	Stabiliser (numerals refer to the ortho	previoualy)	v oxidized polyethylene (OPEX 660 MI)	>	>	trimethylol propane diallyl ether	>		dimethyloctadiynediol (DMOD)	VI	VIII	III	VI	XVII	X	V TMPA
		Run	13	14	15		16			17	18	61	20	21	22	23

	1														
ourless wn)	20 min.	0.7											7.0		
0 = Colourless 15 = Brown)	15 min.	0.7				1.5	1.2	0.5				3.0	2.5	2.5	1.8
	10 min.	0.7			2.3	1.0	1.2	0.5	1.8	0.5	2.0	2.5	2.5	2.2	1.2
Colour (Gardner Scale	4 min.	0.7			2.3	1.0	1.2	0.5	1.0	0	2.8	2.3	1.2	2.2	1.2
Colo	2 min.	0.7			2.3	1.0	1.2	0	1.0	0	2.2	2.0	0.8	2.2	1.2
Polymer	(°C.)	180—200			180—200	176—196	182—204	170—188	170—188	172—188	182—199	186—203	180—200	170200	160-200
£	1 orque (Kg)	1.1			1:1	1.2	1.8	1.1	1.2—1.8	1.4	1.2	1.3—	1.2—	1.1	1.1
Decompos- ition Time	after Flux (min.)	25.5			12	16	18	70	12.5	11	11	17	21	15	16
Flux	Time Min.	1.0			1.0	1.0	1.0	2.0	1.5	1.0	1.0	1.5	1.0	0.7	2.5
Concentra- tion (parts	per hundred of polymer)	5.0	2.0	СН	5.0	2.0	5.0	5.0	5.0	5.0 2.0	5.0	5.0	5.0	5.0	5.0
Stabiliser (numerals refer to the ortho	esters described previously)	Λ	сн,сн,-с,-(сн,он),	CH2OCH2CH2—CH3	×	X (TMPA)	XVIII	XVIII (TMPA)	XI	XI (TMPA)	XIX	VII	VII (TMPA)	ХІІ	XII
	Run	24	J		25	26	27	78	53	30	31	32	33	34	35

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		louriess wa)	20 min.		2.0	0.5					2.0				
		0 = Colouri 15 = Brown)	15 min.		2.0	9.0	8.0				2.0	2.0			
		Colour (Gardner Scale 0 = Colourless 15 = Brown)	10 min.	15	1.0	9.0	8.0	9.0			2.0	3.0	2.5		
		ur (Gardr	4 min.	1.5	9.0	0.5	8.0	0.5	0.3	0.3	3.0	3.0	3.0	2.5	2.8
		Color	2 min.	1.5	0.2	0.5	0.5	0.3	0.3	0.3	2.0	2.0	2.5	2.0	2.5
		Polymer	(°C.)	182—198	180—196	178—200	180—199	182—200	180—196	180—196	182—197	190—198	178—202	186—196	188—192
TABLE—continued	ontinued	8	lorque (Kg)	1.2	1.3	1.8					1.2	1.1	1.2		1.2
	TABLE-c	Decomposition Time	after Flux (min.)	13 (ill defined)	21	22.0	18.5	13.0	8.0	9.5	1.9	17	10	7.5	3.0
			Time Min.	1.0	3.0	1.0	1.0	1.0	1.5	1.0	2.0	2.0	2.0	2.0	2.0
		Concentra- tion (parts	per hundred of polymer)	5.0	5.0 1.0 1.0	5.0	4.0 2.0	3.0	2.0	3.0	5.0	4.0	3.0	2.0	1.0
		Stabiliser (numerals refer to the ortho	esters described previously)	I	V (TMPA) (DMOD)	V (TMPA)	V (TMPA)	v (TMPA)	V (TMPA)	V (TMPA)	>	>	>	>	>
			Run	36	37	38	39	40	41	42	43	4	45	4	47

	ourless wn)	20 min.												12
	0 = Colourless 15 = Brown)	15 min.		>15	1.8					0.5			8.0	4.0
	Colour (Gardner Scale	10 min.	black at 7.5 min.	>15	1.8	1.8				0.5	1.5		5.0	4.0
	ur (Gardı	4 min.	15	3.0	1.8	1.8	1.8	3.0	2.0	0.5	1.0	0.2	4.5	1.5
	လို	2 min.	8	2.5	1.8	1.8	1.8	2.5	1.0	0.5	0.5	0	1.5	1.0
	Polymer T	(°C.)	201—212	190208	190—203	194—203	193—201	194—200	180—188	190—206	190—200	186—198	190—199	188—206
ontinued	£	(Kg)	1.85	1.8	1.3	1.1	1.4	1.3	1.3	1.3	1.3	1.1—	1.1—	1.1
TABLE—continued	Decompos-	(min.)	4.5	9.0	14.0	9.0	6.5	4.5	2.0	15.5	11.0	4.5	16	19
	Flux	Min.	3.0	3.0	2.0	3.0	2.5	2.5	4.0	2.0	2.5	3.5	2.0	2.0
	Concentra- tion (parts	of polymer)		10.0	5.0	4.0	3.0	2.0	1.0	4.0	3.0	3.0	5.0	5.0
	Stabiliser (numerals refer to the ortho	esters described previously)	None	XIII	ΛX	XV	xv	XV	XV	XV Benzyl alcohol	XV Benzyl alcohol	XV Benzyl alcohol	ΧX	XX
		kun	48	49	20	51	22	53	54	55	26	57	28	59

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We make no claim herein to the above-mentioned compositions claimed in Specification No. 1,110,958.

Subject to the foregoing disclaimer,

WHAT WE CLAIM IS: -

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1. Heat stabilized compositions which comprise vinyl chloride homopolymers or copolymers and, as stabilizer, at least one compound of formula:

A¹
0
R-C-0-A¹

wherein R represents an alkyl, phenyl, phenylalkyl, alkylphenylalkyl, halophenyl, nitrophenyl or alkenyl radical and A¹, A², and A³ which may be the same or different, are hydrocarbon radicals or oxygen-interrupted hydrocarbon radicals and wherein any two or all of A¹, A² and A³ may be joined together to form a ring or rings, or of formula:

x-(R⁴--C-OR²)₂

wherein R¹, R² and R², which may be the same or different, represent alkyl, phenyl, phenylalkyl, alkylphenyl, epoxyalkyl or alkylphenylalkyl radicals, R⁴ represents an alkylene, phenylene or alkylphenylene radical and X represents an oxygen or sulphur atom or a single bond.

2. Compositions according to claim 1, containing from 1 to 10% of stabilizer by weight based on the weight of the polymer.

3. Compositions according to claim 1 or 2, wherein the stabilizer is an ortho ester of the first formula specified in claim 1, wherein A¹, A² and A³, represent alkyl, phenyl, phenylalkyl, alkylphenyl, epoxyalkyl or alkylphenylalkyl radicals.

4. Compositions according to claim 1 or 2, wherein the stabilizer is an ortho ester of the formula:

wherein R³ represents an alkylene, phenylene, alkylalkylene, alkenylene, alkenyloxyalkyl alkylene or alkynylene radical, n is an integer from 1 to 4 and R is as defined in claim 1 and A² and A² are as defined in claim 3.

Compositions according to claim 1 or 2, wherein the stabilizer is an ortho ester of the formula:

wherein R^{ϵ} represents an alkylene, alkylene or alkenylalkoxy alkylene radical and R is as defined in claim 1 and A^{ϵ} as defined in claim 3.

6. Compositions according to claim 1 or 2, wherein the stabilizer is an ortho ester of the formula:

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$$0 \\ R - C - 0 - (R^{5}0)_{R} \\ C - R^{6}$$

wherein R is as defined in claim 1, R⁵ and n are as defined in claim 4 and R⁶ is as defined in claim 5.

7. Compositions according to claim 1 or 2, wherein the stabilizer is an ortho ester of the formula:

$$R - C - 0 - R^{7} - C - R^{8}$$

$$0 - R^{7} - C - R^{8}$$

wherein R' represents an alkylene radical, R is as defined in claim 1, and R⁸ has any of the meanings given for R in claim 1.

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8. Compositions according to claim 1 or 2, wherein the stabilizer is an ortho ester of the formula: 10

wherein y is an integer of from 2 to 10.

9. Compositions according to any one of the preceding claims, which comprise also from 0.1 to 10 moles of a high boiling alcohol per mole of ortho ester.

10. Compositions according to claim 9, wherein the alcohol has a boiling point

11. Compositions according to claim 9 or 10, wherein the alcohol is o-, m- or above 175°C.

p- xylene α,α'-diol, trimethylolpropane monopropyl ether, trimethylol propane monoallyl ether, propylene glycol, diethylene glycol, dimethyloctadiynediol, pentaerythritol, trimethylolpropane, neopentylglycol, benzyl alcohol or dipentaerythritol.

12. Compositions according to claim 1, substantially as hereinbefore described.

13. A method of preparing heat-stabilized compositions as claimed in any one of the preceding claims, which comprises preparing a slurry of finely divided vinyl chloride homopolymer or copolymer in a solution of the ortho ester in a solvent therefor and removing the solvent to obtain polymer particles coated with the ortho ester

14. A method according to claim 13, substantially as hereinbefore described. J. A. KEMP & CO.,

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L27 ANSWER 44 OF 48 CAPLUS COPYRIGHT 2003 ACS
   1969:492299 CAPLUS Full-text
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    Heat stabilized poly(vinyl chloride)
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KIND DATE APPLICATION NO. DATE

P 19690507 GB 19660801

Ortho esters and, optionally, high-boiling alcs. were used as nontoxic heat stabilizers for vinyl chloride homopolymers and copolymers. Thus, poly-(vinyl chloride) (I) having a number-average mol. weight of 38,000 was dry blended with 5% oxydiethylene bis(ethylene cyclic orthoacetate) (II) on a Brabender Plastograph at 190°. The time required to form a workable plastic mass (flux time) was 2.0 min., the force required to work the plastic mass (average torque value) was 1.25 kg., the crosslinking or decomposition time was 20.0 min., and the polymer temperature was 199-204°. Rigid moldings produced from the polymer blend had a low degree of color change and good clarity. The addition of 0.1-10 moles high-boiling alc./mole ester enhanced the effect of the ester. When I was blended with 5% II and 2% m-xylene- α,α' -diol, the time required to form a workable plastic mass was 1.5 min., the force required to work the plastic mass was 1.25 kg., the crosslinking or decomposition time was 26.0 min., and the polymer temperature was 193-9°. Similar heat-stabilized compns. were prepared using propylene bis(di-Et orthoacetate), 2-butenylene bis-(di-Et orthoacetate), 1methyltrimethylene bis(1-methyltrimethylene cyclic orthoacetate), propylene bis(propylene cyclic orthoacetate), trimethylolpropane bicyclic orthoacetate, 2-(allyloxymethyl) -2-ethyltrimethylene bis[2- (allyloxymethyl)-2-ethyltrimethylene cyclic orthoacetatel], hexapropyl orthoadipate, triglycidyl ortho-acetate, 2,2dimethyltrimethylene bis(2,2-dimethyltrimethylene cyclic orthoacetate), poly[oxy(2methyldioxolane - 2,4-diyl)-methylene], tri-Et orthoacetate, hexa-Me orthoadipate, 2-(allyloxymethyl)-2-ethyl - 1,2-butanediyl bis(di-Et orthoacetate), ethylene bis(ethylene cyclic orthoacetate), or ethylene bis(2,2-di-methyltrimethylene cyclic orthoacetate). Addnl. alcs. used were m- and p-xylene- α , α '-diol, trimethylolpropane mono-Pr ether, trimethylolpropane monoallyl ether, propylene glycol, diethylene glycol, dimethyloctadiynediol, pentaerthyritol, trimethylolpropane, neopentyl glycol, benzyl alc., and dipentaerythritol.

IT 57-55-6, uses and miscellaneous

RL: USES (Uses)

PATENT NO.

(heat stabilizers from ortho esters and, for chloroethylene polymers)

IT 9002-86-2, uses and miscellaneous

RL: USES (Uses)

(stabilizers for, alcs. -ortho esters as)